



**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Applicants : **Bernard BUGNET, Max COSTA and Denis DONIAT**

Serial No. : **08/691,241** GAU: 1741

Filed : **August 2, 1996** Examiner: **E. WONG**

For : **Porous Structures Having a Pre-metallization  
Conductive Polymer Coating and Method of  
Manufacture**

**DECLARATION**

S. C. P. S. Société de Conseil et de Prospective Scientifique S.A., whose business address is 49 Avenue Ledru Rollin, F-94170 Le Perreux-sur-Marne, FRANCE, is the Assignee of the above-identified patent application filed August 2, 1996 as shown by the records in the Patent and Trademark Office and hereby makes the following declaration.

**COMPARATIVE TESTS**

In order to support their argumentation, the inventors/assignee have carried out a comparative test between the process disclosed in JP 6-248491 and the process of the invention.

For each test, the structure to be metallized is an open-pored polyurethane foam, manufactured by RECTICEL, of 90 PPI grade, 2.2 mm thick and 10 x 25 cm dimensions and whose density is 50g/m<sup>2</sup>.

**Treatment according to JP 6-248491:**

The oxidizing pre-treatment step is carried out by immersing the foam for 4 hours at an ambient temperature in an aqueous solution of ferric chloride having a concentration of 15 g/l. The foam is then drained and placed in a closed vat containing 10 cm<sup>3</sup> of pyrrole. The foam is suspended from the vat top without being in contact with the liquid pyrrole.

After 3 hours, the foam is extracted and then washed and dried under 60°C for 3 hours

The quantity of pyrrole, which is deposited on the foam, is 6 g/m<sup>2</sup>. The deposit is not properly attached to the foam. Measurement of the resistance of the foam gives values comprised between 150 and 250 Ω square. A microscope observation shows that the deposit is not continuous.

Electrolytic deposition of nickel is carried out in a sulfamate bath at 50°C, the electrical contact surface is 10 x 0.5 cm, and the current is led in by one of the foam's width. The foam is suspended by the electrical contact and then placed between two nickel anodes separated by 3 cm from each other.

Electrolysis conditions are the following : current 10 V, time for electrical intensity to reach 40 A, holding this intensity during 5 minutes.

Results: The electrical intensity of 40 A could not be reached after 5 minutes of current application. The intensity reached 12 A only.

The average nickel deposit was 4 cm height and shows characteristic arborescent reliefs of a deposit over a non-homogenous and weakly conductive structure.

Treatment according to the invention :

The oxidising pre-treatment step is carried out by immersing the foam for 5 minutes at 65°C in an aqueous solution of KMnO<sub>4</sub> having a concentration of 20 g/l and pH 7. The foam is then rinsed with water for 30 seconds and dried under hot air (50°C) for 2 minutes.

The quantity of MnO<sub>2</sub> deposited on the foam is 2.8 g/m<sup>2</sup> of the foam.

The step of fixing the monomer is carried out by immersing the foam in an aqueous solution of pyrrole as follows :

- 50 cm<sup>3</sup>/litre of pyrrole,

- 50 cm<sup>3</sup>/litre of isopropanol,

- 900 cm<sup>3</sup>/litre of water.

The foam immersion time is 6 minutes and the temperature of the solution is 18°C.

The foam is drained for 60 seconds and then immersed in an aqueous solution containing :

- 80 g/l of ferric chloride,

- 35 g/l of fluoroboric acid,

at 20°C for 10 minutes.

The foam is rinsed with water and dried under hot air for 3 minutes. The quantity of pyrrole deposited on the foam is 8 g/m<sup>2</sup> of the foam.

Measurement of the resistance on many parts of the foam gives values comprised between 15 and 22 Ω square. A microscope observation shows that the polypyrrrole deposit is homogenous and perfectly continuous on the surface of the fibers. The foam is slightly stiffened.

Electrolytic deposition of nickel is carried out in a sulfamate bath at 50°C, the electrical contact surface is 10 x 0.5 cm and the current is led in by one of the foam's width. The foam is suspended by the electrical contact and then placed between two nickel anodes separated by 3 cm from each other.

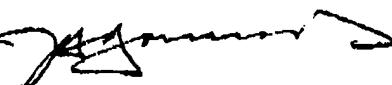
Electrolysis conditions are the following : current 10 V, time for electrical intensity to reach 40 A, holding this intensity during 5 minutes.

Results : The electrical intensity of 40 A was reached after 22 seconds of current application, then held for 5 minutes.

The whole surface of the foam was coated with nickel.

The undersigned, Jacques DONLAT, declares that he is the President of the Executive Board of Assignee association and is authorized to execute this Declaration on behalf of said Assignee; he declares that all of the statements made herein of his own knowledge are true, and that all statements made herein on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements or the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and may jeopardise the validity of this application or any patent issuing therefrom.

S. C. P. S. Société de Conseil et de Prospective Scientifique S.A.

April 30th, 2001   
Date

President of the Executive Board  
(Title)